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(54) SAFETY GLASS

(57)Abstract:
PURPOSE: To obtain safety glass effectively shielding heat ray without dropping a visible light transmittance, producible without changing the conventional production process of safety glass, by laying a non-rigid resin layer containing a heat ray shielding metal oxide between a pair of glass plates.
CONSTITUTION: In safety glass comprising a pair of glass plates and a non-rigid resin layer laid between a pair of glass plates, a heat ray shielding metal oxide is added to the non-rigid resin layer. SnO2 or In2O3 having ≥0.1μm particle diameter is especially suitable as the heat ray shielding metal. The reason why the particle diameter is 0.1μm is that the scattering and absorption of visible light are enlarged and transparency can not be obtained when the particle diameter exceed 0.1μm. It is known that the light scattering by particles is maximized when the particle diameter is a size of 1/2 wavelength and is proportional to the sixth power of particle diameter in the region smaller than it.

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CLAIMS

[Claim(s)]
[Claim 1] It is the glass laminate characterized by this elasticity resin layer containing the heat ray electric shielding nature metallic oxide in the glass laminate which consists of an elasticity resin layer prepared between the glass of a pair, and this glass.
[Claim 2] The glass laminate according to claim 1 with which said heat ray electric shielding nature metallic oxide is characterized by being either the tin oxide with a particle size of 0.1 micrometers or less or indium oxide.
[Claim 3] As a compounding ratio of said heat ray electric shielding nature metallic oxide, it is 0.4 g/m2. Glass laminate according to claim 1 or 2 characterized by blending so that it may become the above.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the glass laminate with which the elasticity resin layer contains the heat ray electric shielding nature metallic oxide in more detail about a glass laminate with the heat ray electric shielding reproductive function.
[0002]
[Description of the Prior Art] The glass laminate which pasted up the glass plate of a pair in the elasticity resin layer, and prevented scattering of the fragment at the time of damaging is used for vehicle windowpanes and structural windowpanes, such as an automobile, a railroad, and an aircraft, crime prevention glass, etc. The approach using heat reflective glass as an approach of giving the heat ray electric shielding engine performance to a glass laminate, the approach of mixing organic dye in an elasticity resin layer, and using as a coloured film, the method of using a heat ray reflective film for an elasticity resin layer, etc. are performed conventionally.
[0003] As an approach by heat reflective glass, the glass which carried out the laminating of tungstic oxide and the silver thin film by the vacuum deposition method, the sputtering method, etc. is used for the publication-number 6-No. 144891 official report among the above-mentioned conventional techniques, for example. Moreover, by scouring organic dye to resin, such as a polyvinyl butyral, and coloring it Green, blue, Brown, etc., the approach by coloring of organic dye makes a part of visible ray absorb, and is reducing the heat energy of sunlight.
[0004] The approach by the heat ray reflective film carries out the laminating of silver and the oxide thin film, fastens them by the polyvinyl butyral, and is making for example, a Provisional-Publication-No. 56-No. 3252 official report and a Provisional-Publication-No. 63-No. 134332 official report rival glass by vacuum evaporation technique, the sputtering method, etc. in plastic film, such as polyester, further again. And in the Provisional-Publication-No. 60-No. 127152 official report and the publication-number 6-No. 191906 official report, the laminating of a heat ray reflecting layer or a film, and the coating layer or film of a near infrared ray absorbent is carried out, and the electric shielding engine performance is raised.
[0005]
[Problem(s) to be Solved by the Invention] By the approach of giving the heat ray electric shielding engine performance to the glass laminate in the former For example, in order to use a vacuum deposition method and the sputtering method by the approach using heat reflective glass, It is inferior to productivity, and there is a trouble of becoming cost quantity, and an elasticity resin layer by the approach of mixing organic dye and using as a coloured film Light permeability falls and it becomes very inconvenient in the application aiming at coloring. By the approach of organic dye having the trouble that lightfastness is bad and does not bear prolonged use, and using a heat ray reflective film for an elasticity resin layer In order to use a vacuum deposition method and the sputtering method for production of the heat ray reflective film, it is inferior to productivity, there is a trouble of becoming cost quantity, and, moreover, the trouble that the manufacture approach of the conventional glass laminate only using polyvinyl butyral resin must be changed is also produced.
[0006] This invention was made in view of such a trouble, and it can be made, without changing the production process of the conventional glass laminate in any way, and it is going to offer the glass laminate which covers a heat ray effectively, without reducing light permeability.
[0007]

[Means for Solving the Problem] It found out that the glass laminate which absorbs a heat ray effectively, without not changing the production process of the conventional glass laminate at all, but reducing visible-ray permeability into the elasticity resin layer used for a glass laminate by distributing to homogeneity the particle which does not absorb a visible ray but absorbs a heat ray when this invention persons inquire wholeheartedly in view of said trouble was made, and resulted in this invention.

[0008] That is, in the glass laminate with which the glass laminate according to claim 1 in this invention consists of an elasticity resin layer prepared between the glass of a pair, and this glass, it is characterized by this elasticity resin layer containing the heat ray electric shielding nature metallic oxide.

[0009] And as for a glass laminate according to claim 2, it is desirable that it is characterized by said heat ray electric shielding nature metallic oxide being either the tin oxide with a particle size of 0.1 micrometers or less or indium oxide.

[0010] And for a glass laminate according to claim 3, said heat ray electric shielding nature metallic oxides are 0.4 g/m2. Blending so that it may become the above is desirable.

[0011] [Concrete explanation of invention] This invention is explained in more detail hereafter. This invention is attained by distributing a heat ray electric shielding nature metallic oxide to homogeneity in the elasticity resin which constitutes the interlayer of a glass laminate.

[0012] All can be used for it, if the heat ray electric shielding nature metallic oxide used in this invention has small absorption of the light as much as possible and its 780-2500nm [it is usually considered that is a heat ray] absorption is large. as such a thing -- SnO2, In 2O3, CdO, Cd2SnO4, FeO, Fe 3O4, ZnO, VO2, and V2O5 etc. -- a transparent conductive oxide can be mentioned.

[0013] It sets especially to this invention and particle size is SnO2 0.1 micrometers or less. Or In 2O3 It is suitable. As a reason for setting particle size to 0.1 micrometers, when it exceeds 0.1 micrometers, it is because dispersion of the light and absorption become large and transparency is no longer acquired. As for dispersion of the light by the particle, it is known that in the case of one half of the magnitude of wavelength particle size serves as max and is proportional in the range smaller than it at the 6th power of particle size.

[0014] When the metallic oxide with which particle size differed was distributed, the distributed particle size and transparency of the light were investigated and distributed particle size was 0.1 micrometers or less, this invention persons found out becoming transparent substantially, and resulted in this conclusion. Furthermore, particle size is so desirable that it is small, and is 0.05 micrometers or less more preferably. SnO2 used for this invention for the above-mentioned reason Or In 2O3 0.1 micrometers or less, if particle size is 0.05 micrometers or less more preferably, especially a limit will not have it.

[0015] And SnO2 As the manufacture approach, the approach of a publication can be mentioned into a publication-number 2-No. 105875 official report. Doping a different-species element in order to raise the heat ray electric shielding engine performance does not interfere. As a dopant, Sb, P, Te, W, Cl, and F are suitable.

[0016] Moreover, In 2O3 Filtration washing of the sludge which neutralized and was able to do the water solution of indium salts, such as indium chloride, a nitric-acid indium, and indium sulfate, with alkali, such as NaOH and NH4OH, as the manufacture approach can be carried out, and the approach of calcinating by reducing atmosphere further can be illustrated. Doping a different-species element in order to raise the heat ray electric shielding engine performance does not interfere. As a dopant, Sn, Mo, Zr, Ti, Sb, W, and F are suitable.

[0017] As elasticity resin used for the glass laminate of this invention, although polyvinyl butyral resin is chiefly used abundantly, even if it is other resin, it does not interfere. For example, resin liquefied between the glass plates of two sheets is set to the so-called impregnation type which carried out impregnation hardening of glass laminate, and it is to liquefied resin. A heat ray electric shielding nature metallic oxide can be distributed.

[0018] That is, after this invention applies to glass of one of the two the glass laminate which stretched the glass plate of two sheets with the polyvinyl-butyral-resin film, and the solution of polyvinyl butyral resin and forms membranes, it can be used for the glass laminate on which another glass was pasted up further, the glass laminate which is made to pour in and harden liquefied resin, such as an acrylic, between the glass of two sheets, and is made.

[0019] As an approach of distributing a heat ray electric shielding nature metallic oxide to elasticity resin, the conventional distributed approach is employable. That is, it is possible to use distributed equipments, such as a ball mill, a sand mill, and attritor. At this time, it is desirable to use dispersants which do not have a bad influence on the engine performance of elasticity resin, such as a surfactant and a high molecular compound.

Moreover, when using additives, such as a solvent, a plasticizer, a stabilizer, and a coloring agent, for elasticity resin, it is also possible to distribute on an additive beforehand and to mix with resin behind. this approach -- ordinary temperature -- solid resin -- although it is liquefied, it is effective especially when energy with it is required. [high viscosity and] [to distribution] [big]

[0020] For example, as an approach of making polyvinyl butyral resin distributing a heat ray electric shielding nature metallic oxide, it can distribute to plasticizers, such as dioctyl phthalate, tributyl phosphate, octyl diphenyl phosphate, cresyl diphenyl phosphate, and a dibutyl sebacate, and the approach of making this a film according to the shaping approach of conventional polyvinyl butyral resin can be adopted first.

[0021] At this time, as a rate of a compounding ratio of the heat ray electric shielding nature metallic oxide to a plasticizer, considering as 10 - 400% is desirable, and it is 0.4g/m2 by the electric shielding engine performance as concentration in polyvinyl butyral resin. Blending so that it may become the above is desirable. The reason is 0.4g/m2. If it does not fill, the difference of solar radiation permeability and light permeability becomes less than 5%, and it is because the shielding effect is small. Moreover, when dissolving polyvinyl butyral resin with a solvent and it is liquefied, in the case of the liquefied resin for impregnation, such as acrylic resin, the approach of distributing directly can be taken.

[0022] As an approach of making a glass laminate, it is more possible than the film or solution of elasticity resin which distributed the heat ray electric shielding nature metallic oxide to adopt the manufacture approach of the conventional glass laminate as it is. For example, in the thing using a polyvinyl-butyral film, a polyvinyl-butyral film is put between the glass plates of two sheets, preliminary adhesion is carried out whenever [glass temperature / of 80-100 degrees C /, and reduced pressure] at 650 or more mmHgs, and, subsequently they are the temperature of 120-150 degrees C, and the pressure of 10-15kg/cm2. By performing this adhesion for 20 - 40 minutes in an autoclave, it can consider as a glass laminate.

[0023] As a glass material used for the glass laminate of this invention, if it uses for the object for construction, or vehicles, all are usable and can usually use plate glass, a float glass, heat absorbing glass, etc. Especially in the glass laminate of this invention, it is suitable, and heat absorbing glass can migrate to a near-infrared full wave length region, and can obtain the high electric shielding engine performance.

[0024]

[Example] Next, an example explains this invention in more detail.

(Example 1)

[Production of SnO2] SbCl3 of the 46.2 weight sections Dissolved SnCl4 and 5H2O of the 670 weight sections in the 6N-HCl solution of the 3000 weight sections, 25% of ammonia liquor 2000 weight section was made to add and react to this, sol-like dispersion liquid were obtained, and filtration washing was carried out until it became impossible for the ammonium chloride to have detected this.

[0025] Subsequently, after heating this at 350 degrees C and holding it with a well-closed container for 5 hours, the steam was emitted by the cooling process, it condensed to 25 % of the weight of solid content, and antimony content tin-oxide (it is hereafter written as ATO) dispersion liquid of 50A of mean diameters were obtained. Ten weight sections addition of the octadecyl amine was carried out at this dispersion-liquid 400 weight section, and the coagulation sedimentation object was obtained. This aggregate was taken out by filtration, it dried at 100 degrees C for 2 hours, and ATO powder was obtained.

[0026] [Production of dispersion liquid] Said ATO powder 36.3 weight section and the toluene 73.7 weight section were mixed, and the ultrasonic disperser distributed for 5 minutes. Three weight sections addition of the 10% toluene solution of an anion system surface active agent could be carried out at these dispersion liquid, it mixed, and 114 weight sections mixing of the dioctyl phthalate was carried out further. Vacuum distillation of this mixed liquor was carried out for 15 minutes at 95 degrees C, and toluene was removed. This liquid is the liquid which ATO distributed 20% to dioctyl phthalate at homogeneity.

[0027] [Production of an interlayer and a glass laminate] The film of a polyvinyl butyral with the ATO content of about 8.4g/ma 2, and a thickness of 0.76mm was produced for the above-mentioned dispersion liquid to the polyvinyl-butyral-resin 100 weight section 5 weight sections and by mixing 35 weight sections and the ultraviolet ray absorbent (Ciba-Geigy make trade name: tinuvin P) 0.15 weight section, making it scour each other's dioctyl phthalate enough further, and carrying out extrusion molding of this. Subsequently, the obtained film is inserted with 3mm sheet glass, and they are the glass temperature of 70 degrees C, and the pressure of 5kg/cm2. It pastes up and they are the temperature of 135 degrees C, and the pressure of 12kg/cm2 further. It

pressed with the autoclave and the glass laminate was produced. The light permeability of this glass laminate is 73.9%, and solar radiation permeability is 57.0%. The optical property of the produced glass laminate is shown in drawing 1 with the optical property of the conventional glass laminate (3mm float glass +0.76mm polyvinyl-butyral +3mm float glass). As shown all over drawing, that whose conventional glass laminate was 100% decreased sharply, permeability became 1/2 or less [conventional by about 900nm or more], and it became 1/3 or less [conventional by about 1100nm or more].

[0028] (Example 2)

[Production of In 2O3] White precipitate was made to generate in addition until it dissolved the indium chloride 154.5 weight section and the stannic-chloride 5.2 weight section in the pure-water 2000 weight section, it considered as the homogeneity solution and pH was set to 12 to this in aqueous ammonia, and it filtered and washed until it became impossible to have detected the ammonium chloride.

[0029] Pure water was added and it considered as the shape of a slurry so that it might become the precipitate of a cleaning agent with 5 % of the weight of solid content, and it dried with the spray dryer and considered as white powder. It is the obtained white powder further for 5 minutes at 450 degrees C among atmospheric air N2 H2 275 degrees C was calcinated for 40 minutes in mixed gas. The obtained powder is indium oxide (following, ITO) to which the tin oxide dissolved [particle size] 3% of the weight by 0.03 micrometers.

[0030] [Decomposition to a plasticizer] 30 weight sections were carried out for said ITO powder, 3 weight sections mixing of 70 weight sections and the anion system surfactant was carried out for dioctyl phthalate, and the Sand grinder distributed for 3 hours.

[0031] [Scour lump by resin] The film of a polyvinyl butyral with the ITO content of about 5.0g/ma 2, and a thickness of 0.76mm was produced for the above-mentioned dispersion liquid to the polyvinyl-butyral-resin 100 weight section 2 weight sections and by mixing 38 weight sections and the ultraviolet ray absorbent (Ciba-Geigy make trade name: tinuvin P) 0.15 weight section, making it scour each other's dioctyl phthalate enough further, and carrying out extrusion molding of this. In this way, the obtained film is inserted with 3mm sheet glass, and they are the glass temperature of 70 degrees C, and the pressure of 5kg/cm2. It pastes up and they are the temperature of 135 degrees C, and the pressure of 12kg/cm2 further. It pressed with the autoclave and the glass laminate was produced. The light permeability of this glass laminate is 84.6%, and solar radiation permeability is 65.2%. The optical property of the produced glass laminate is shown in drawing 1 . As shown all over drawing, permeability became 1/2 or less [conventional by about 1050nm or more], and became 0% substantially in 1500nm or more.

[0032] (Example 3) In this example 2, the case where one in the glass of a pair was used as heat absorbing glass was produced according to the same manufacture process as an example 2. The light permeability of this glass laminate is 65.2% of solar radiation permeability 77.5%. In this case, the optical property of the glass laminate which can be set is shown in drawing 1 . As shown all over drawing, it became 1/2 or less [conventional by about 850nm or more], and permeability decreased or less [conventional] to 1/3 in 900nm - 1400nm, and became 0% substantially in 1400nm or more.

[0033]

[Effect of the Invention] Therefore, in the glass laminate according to claim 1 in this invention, the glass laminate which has heat ray electric shielding nature by low cost can be realized, without changing and adding the manufacture approach of the conventional glass laminate in any way, since the elasticity resin layer prepared between the glass of a pair and this glass contains the heat ray electric shielding nature metallic oxide.

[0034] And with a glass laminate according to claim 2, since said heat ray electric shielding nature metallic oxide is either the tin oxide with a particle size of 0.1 micrometers or less or indium oxide, heat ray electric shielding nature can realize a good glass laminate again that there is little absorption of the light as much as possible.

[0035] And with a glass laminate according to claim 3, they are 0.4 g/m2 again as a compounding ratio of said heat ray electric shielding nature metallic oxide. Since it blended so that it might become the above, the difference of visible-ray permeability and solar radiation permeability can make it to 5% or more.

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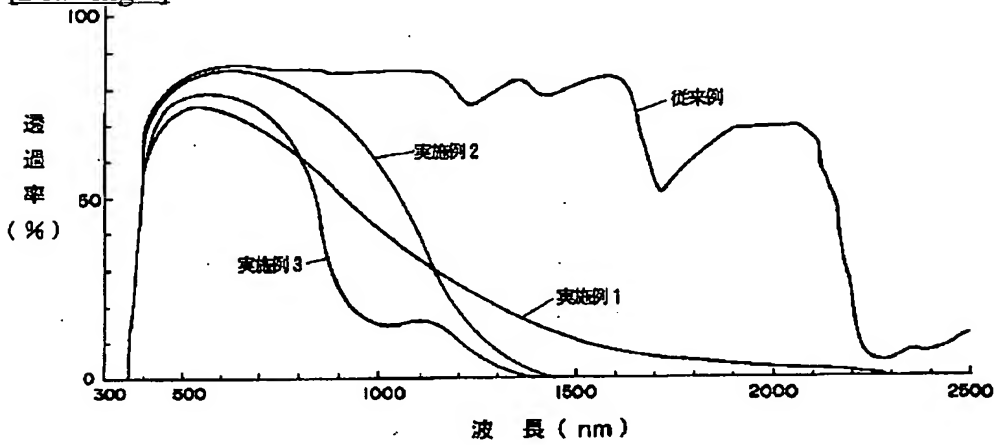
DRAWINGS

[Drawing 1]



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CONSTITUTION: In safety glass comprising a pair of glass plates and a non-rigid resin layer laid between a pair of glass plates, a heat ray shielding metal oxide is added to the non-rigid resin layer. SnO2 or In2O3 having ≥0.1μm particle diameter is especially suitable as the heat ray shielding metal. The reason why the particle diameter is 0.1μm is that the scattering and absorption of visible light are enlarged and transparency can not be obtained when the particle diameter exceed 0.1μm. It is known that the light scattering by particles is maximized when the particle diameter is a size of 1/2 wavelength and is proportional to the sixth power of particle diameter in the region smaller than it.

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	27/18				27/18	Ｚ
// Ｂ６０Ｊ	1/00			Ｂ６０Ｊ	1/00	Ｈ
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審査請求　未請求　請求項の数３　ＯＬ　（全 5 頁）

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(54)【発明の名称】　合わせガラス

(57)【要約】

【目的】　熱線遮蔽性機能をもった合わせガラスに関し、従来の合わせガラスの製造工程を何ら変更することなく作ることができ、可視光透過率を低下させず熱線を効果的に遮蔽することができるようにすることを目的とする。

【構成】　一対のガラスと該ガラスの間に設けた軟質樹脂層とからなる合わせガラスにおいて、該軟質樹脂層は熱線遮蔽性金属酸化物を含有しているように構成する。

1

【特許請求の範囲】

【請求項１】一対のガラスと該ガラスの間に設けた軟質樹脂層とからなる合わせガラスにおいて、該軟質樹脂層は熱線遮蔽性金属酸化物を含有していることを特徴とする合わせガラス。

【請求項２】前記熱線遮蔽性金属酸化物が、粒径０．１μｍ以下の酸化スズまたは酸化インジウムのいずれかであることを特徴とする請求項１記載の合わせガラス。

【請求項３】前記熱線遮蔽性金属酸化物の配合比として、０．４ｇ／m^2 以上となるように配合することを特徴とする請求項１または２記載の合わせガラス。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、熱線遮蔽性機能をもった合わせガラスに関し、更に詳しくは、軟質樹脂層が熱線遮蔽性金属酸化物を含有している合わせガラスに関する。

【０００２】

【従来の技術】一対のガラス板を軟質樹脂層で接着し、破損した際の破片の飛散を防止した合わせガラスが自動車、鉄道、航空機等の乗り物窓ガラスや建築用窓ガラス、防犯ガラス等に用いられている。合わせガラスに熱線遮蔽性能を付与する方法として、熱線反射ガラスを用いる方法、軟質樹脂層に有機染料を混入して着色フィルムとする方法、軟質樹脂層に熱線反射フィルムを用いる方法等が従来より行われている。

【０００３】上記従来技術のうち、熱線反射ガラスによる方法としては、例えば、特開平６　１４４８９１号公報には、真空蒸着法、スパッタリング法等により酸化タングステンおよび銀薄膜を積層したガラスを用いている。また、有機染料の着色による方法は、ポリビニルブチラール等の樹脂に有機染料を練り込み、グリーン、ブルー、ブラウン等に着色することにより、可視光線の一部を吸収させ太陽光の熱エネルギーを低減している。

【０００４】さらにまた、熱線反射フィルムによる方法は、例えば、特開昭５６　３２５２号公報、特開昭６３　１３４３３２号公報には、ポリエステル等のプラスチックフィルムに真空蒸着法、スパッタリング法等により、銀および酸化物薄膜を積層し、ポリビニルブチラールで挟着し、ガラスと張り合わせている。そして、特開昭６０　１２７１５２号公報、特開平６　１９１９０６号公報では、熱線反射層またはフィルムと、近赤外線吸収剤のコーティング層またはフィルムとを積層し、遮蔽性能を向上させている。

【０００５】

【発明が解決しようとする課題】従来における合わせガラスに熱線遮蔽性能を付与する方法では、例えば、熱線反射ガラスを用いる方法では、真空蒸着法やスパッタリング法を用いるため、生産性に劣り、コスト高となるという問題点があり、軟質樹脂層を有機染料を混入して着色フィルムとする方法では、可視光透過率が低下し、着色を目的としない用途においてははなはだ不都合となり、有機染料は耐光性が悪く、長期間の使用に堪えないという問題点があり、軟質樹脂層に熱線反射フィルムを用いる方法では、熱線反射膜の作製に真空蒸着法やスパッタリング法を用いるため、生産性に劣り、コスト高となるという問題点があり、しかも、ポリビニルブチラール樹脂のみを用いる従来の合わせガラスの製造方法を変更しなければならないといった問題点も生じる。

【０００６】本発明は、このような問題点に鑑みてなされたもので、従来の合わせガラスの製造工程を何ら変更することなく作ることができ、可視光透過率を低下させずに熱線を効果的に遮蔽する合わせガラスを提供しようとするものである。

【０００７】

【課題を解決するための手段】本発明者らは、前記問題点に鑑み鋭意研究を行ったところ、合わせガラスに用いる軟質樹脂層中に、可視光線は吸収せず熱線を吸収する微粒子を均一に分散することにより、従来の合わせガラスの製造工程を何ら変更せず、可視光線透過率を低下させずに熱線を効果的に吸収する合わせガラスができることを見いだし、本発明に至った。

【０００８】すなわち、本発明における請求項１記載の合わせガラスは、一対のガラスと該ガラスの間に設けた軟質樹脂層とからなる合わせガラスにおいて、該軟質樹脂層は熱線遮蔽性金属酸化物を含有していることを特徴とする。

【０００９】そして、請求項２記載の合わせガラスは、前記熱線遮蔽性金属酸化物が粒径０．１μｍ以下の酸化スズまたは酸化インジウムのいずれかであることを特徴とすることが望ましい。

【００１０】そして、請求項３記載の合わせガラスは、前記熱線遮蔽性金属酸化物が０．４ｇ／m^2 以上となるように配合することが好ましい。

【００１１】〔発明の具体的説明〕以下、本発明を更に詳しく説明する。本発明は、合わせガラスの中間膜を構成する軟質樹脂中に熱線遮蔽性金属酸化物を均一に分散することにより達成される。

【００１２】本発明において使用される熱線遮蔽性金属酸化物は、可視光の吸収が極力小さく、通常、熱線と見做されている７８０～２５００ｎｍの吸収が大きいものであれば、いずれも採用可能である。このようなものとしてSnO_2，In_2O_3，CdO，Cd_2SnO_4，FeO，Fe_3O_4，ZnO，VO_2，V_2O_5 等の透明導電性酸化物を挙げることができる。

【００１３】特に本発明においては、粒径が０．１μｍ以下のSnO_2 またはIn_2O_3 が好適である。粒径を０．１μｍとする理由としては、０．１μｍを超えた場合、可視光の散乱、吸収が大きくなり、透明性が得られなくなるからである。粒子による光の散乱は、粒径が波

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長の１／２の大きさの場合に最大となり、それより小さい範囲においては粒径の６乗に比例することが知られている。

【００１４】本発明者らは、粒径の異なった金属酸化物を分散し、その分散粒径と可視光の透明性を調べたところ、分散粒径が０．１μｍ以下であれば、実質的に透明となることを見い出し、本結論に至った。さらに、粒径は小さいほど好ましく、より好ましくは０．０５μｍ以下である。上記の理由により、本発明に用いるSnO_2またはIn_2O_3は、粒径が０．１μｍ以下、より好ましくは０．０５μｍ以下であれば、特に制限はない。

【００１５】そして、SnO_2の製造方法としては、特開平２　１０５８７５号公報中に記載の方法を挙げることができる。熱線遮蔽性能を向上させる目的で異種元素をドーピングすることは差し支えない。ドーパントとしては、Ｓｂ，Ｐ，Ｔｅ，Ｗ，Ｃｌ，Ｆが適している。

【００１６】また、In_2O_3の製造方法としては、塩化インジウム、硝酸インジウム、硫酸インジウム等のインジウム塩の水溶液をＮａＯＨ，NH_4OH等のアルカリで中和してできた析出物を濾過洗浄し、さらに還元雰囲気で焼成する方法を例示することができる。熱線遮蔽性能を向上させる目的で異種元素をドーピングすることは差し支えない。ドーパントとしては、Ｓｎ，Ｍｏ，Ｚｒ，Ｔｉ，Ｓｂ，Ｗ，Ｆが適している。

【００１７】本発明の合わせガラスに用いる軟質樹脂としては、もっぱらポリビニルブチラール樹脂が多用されるが、他の樹脂であっても差し支えない。例えば、２枚のガラス板の間に液状の樹脂を注入硬化させた、いわゆる注入タイプの合わせガラスにおいては、液状樹脂に熱線遮蔽性金属酸化物を分散させることができる。

【００１８】すなわち本発明は、２枚のガラス板をポリビニルブチラール樹脂フィルムで張り合せた合わせガラス、ポリビニルブチラール樹脂の溶液を片方のガラスに塗布して成膜した後さらにもう一方のガラスを接着した合わせガラス、２枚のガラス間にアクリル等の液状樹脂を注入し硬化させて作る合わせガラス等に用いることができる。

【００１９】熱線遮蔽性金属酸化物を軟質樹脂に分散する方法としては、従来の分散方法を採用することができる。すなわち、ボールミル、サンドミル、アトライター等の分散装置を用いることが可能である。このとき、軟質樹脂の性能に悪影響を与えない界面活性剤、高分子化合物等の分散剤を用いることが望ましい。また、軟質樹脂に溶剤、可塑剤、安定剤、着色剤等の添加剤を用いる場合は、予め添加剤に分散し、後に樹脂と混合することも可能である。この方法は、常温で固体の樹脂や、液状であるが粘度が高く分散に大きなエネルギーが必要な場合特に有効である。

【００２０】例えば、ポリビニルブチラール樹脂に熱線遮蔽性金属酸化物を分散させる方法としては、まず、ジオクチルフタレート、トリブチルホスフェート、オクチルジフェニルホスフェート、クレジルジフェニルホスフェート、セバシン酸ジブチル等の可塑剤に分散し、これを従来のポリビニルブチラール樹脂の成形方法に従いフィルムとする方法を採用することができる。

【００２１】このとき可塑剤にたいする熱線遮蔽性金属酸化物の配合比率としては、１０～４００％とすることが望ましく、また、ポリビニルブチラール樹脂中の濃度としては、遮蔽性能により０．４ｇ／m^2以上となるように配合することが望ましい。その理由は、０．４ｇ／m^2に満たないと、日射透過率と可視光透過率の差が５％未満となり、遮蔽効果が小さいからである。また、ポリビニルブチラール樹脂を溶剤で溶解して液状とする場合、およびアクリル樹脂等の液状の注入用樹脂の場合は、直接分散する方法をとることができる。

【００２２】熱線遮蔽性金属酸化物を分散した軟質樹脂のフィルムまたは溶液より合わせガラスを作る方法としては、従来の合わせガラスの製造方法をそのまま採用することが可能である。例えば、ポリビニルブチラールフィルムを用いるものでは、２枚のガラス板の間にポリビニルブチラールフィルムを挟み込み、ガラス温度８０～１００℃、減圧度６５０ｍｍＨｇ以上で予備接着し、次いで、温度１２０～１５０℃、圧力１０～１５ｋｇ／cm^2のオートクレーブ中で２０～４０分間の本接着を行うことにより、合わせガラスとすることができる。

【００２３】本発明の合わせガラスに用いるガラス素材としては、通常、建築用または乗物用等に用いるものであれば、いずれも使用可能で、普通板硝子、フロートガラス、熱線吸収ガラス等を用いることができる。本発明の合わせガラスにおいては、特に熱線吸収ガラスが好適で、近赤外全波長域にわたって、高い遮蔽性能を得ることができる。

【００２４】

【実施例】次に本発明を実施例にてさらに詳しく説明する。

（実施例１）

〔SnO_2の作製〕４６．２重量部の$SbCl_3$と６７０重量部の$SnCl_4 \cdot 5H_2O$を、３０００重量部の６Ｎ－ＨＣｌ溶液に溶解し、これに２５％のアンモニア液２０００重量部を添加して反応させ、ゾル状分散液を得、これを塩化アンモニウムが検出できなくなるまで濾過洗浄した。

【００２５】次いで、これを密閉容器で３５０℃に加熱し、５時間保持した後、冷却過程で水蒸気を放出し、固形分２５重量％まで濃縮し、平均粒径５０Åのアンチモン含有酸化スズ（以下、ＡＴＯと略記する）分散液を得た。この分散液４００重量部にオクタデシルアミンを１０重量部添加し、凝集沈殿物を得た。この凝集物を濾過により取り出し、１００℃にて２時間乾燥しＡＴＯ粉末を得た。

【００２６】〔分散液の作製〕前記ＡＴＯ粉末３６．３重量部とトルエン７３．７重量部とを混合し、超音波分散機により５分間分散した。この分散液にアニオン系界面活性剤の１０％トルエン溶液を３重量部添加して良く混合し、さらにジオクチルフタレートを１１４重量部混合した。この混合液を９５℃で１５分間減圧蒸留し、トルエンを除去した。この液はジオクチルフタレートにＡＴＯが２０％均一に分散した液である。

【００２７】〔中間膜および合わせガラスの作製〕ポリビニルブチラール樹脂１００重量部に対し、上記分散液を５重量部、さらにジオクチルフタレートを３５重量部、および、紫外線吸収剤（チバガイギー社製、商品名：チヌビンＰ）０．１５重量部を混合して十分練り合わせ、これを押出し成形することにより、ＡＴＯ含有量約８．４ｇ／m^2 、厚さ０．７６mmのポリビニルブチラールのフィルムを作製した。次いで、得られたフィルムを３mmの板ガラスで挟み、ガラス温度７０℃、圧力５ｋｇ／ｃm^2 で接着し、さらに温度１３５℃、圧力１２ｋｇ／ｃm^2 のオートクレーブでプレスし、合わせガラスを作製した。この合わせガラスの可視光透過率は７３．９％、日射透過率は５７．０％である。作製した合わせガラスの光学特性を、従来の合わせガラス（３mmフロートガラス＋０．７６mmポリビニルブチラール＋３mmフロートガラス）の光学特性とともに図１に示す。図中に示すように、透過率が従来の合わせガラスが１００％であったものが、大幅に減少し、約９００ｎｍ以上で従来の１／２以下になり、約１１００ｎｍ以上で従来の１／３以下になった。

【００２８】（実施例２）

〔In_2O_3の作製〕塩化インジウム１５４．５重量部、塩化第二スズ５．２重量部を純水２０００重量部に溶解して均一溶液とし、これに、アンモニア水をｐＨが１２になるまで加え、白色の沈殿物を生成させ、塩化アンモニウムが検出できなくなるまで濾過し洗浄した。

【００２９】洗浄剤の沈殿物に固形分５重量％となるように純水を加えてスラリー状とし、スプレードライヤーで乾燥して、白色粉末とした。得られた白色粉末を大気中４５０℃で５分間、さらにN_2 とH_2 の混合ガス中で２７５℃、４０分間焼成した。得られた粉末は、粒径が０．０３μmで酸化スズが３重量％固溶した酸化インジウム（以下、ＩＴＯ）である。

【００３０】〔可塑剤への分解〕前記ＩＴＯ粉末を３０重量部、ジオクチルフタレートを７０重量部、アニオン系界面活性剤を３重量部混合し、サンドグラインダーにより３時間分散した。

【００３１】〔樹脂への練り込み〕ポリビニルブチラール樹脂１００重量部に対し、上記分散液を２重量部、さらにジオクチルフタレートを３８重量部、および紫外線吸収剤（チバガイギー社製、商品名：チヌビンＰ）０．１５重量部を混合して十分練り合わせ、これを押出成形することにより、ＩＴＯ含有量約５．０ｇ／m^2 、厚さ０．７６mmのポリビニルブチラールのフィルムを作製した。こうして得られたフィルムを３mmの板ガラスで挟み、ガラス温度７０℃、圧力５ｋｇ／ｃm^2 で接着し、さらに温度１３５℃、圧力１２ｋｇ／ｃm^2 のオートクレーブでプレスし、合わせガラスを作製した。この合わせガラスの可視光透過率は８４．６％、日射透過率は６５．２％である。作製した合わせガラスの光学特性を図１に示す。図中に示すように、透過率が約１０５０ｎｍ以上で従来の１／２以下となり、１５００ｎｍ以上では実質的に０％となった。

【００３２】（実施例３）この実施例２において、一対のガラスのうち１枚を熱線吸収ガラスにした場合を、実施例２と同様の製造過程により作製した。この合わせガラスの可視光透過率は７７．５％、日射透過率６５．２％である。この場合における合わせガラスの光学特性を図１に示す。図中に示すように、透過率が約８５０ｎｍ以上で従来の１／２以下となり、９００ｎｍ～１４００ｎｍでは従来の１／３以下に減少し、１４００ｎｍ以上では実質的に０％となった。

【００３３】

【発明の効果】従って、本発明における請求項１記載の合わせガラスでは、一対のガラスと該ガラスの間に設けた軟質樹脂層が熱線遮蔽性金属酸化物を含有しているため、従来の合わせガラスの製造方法を何ら変更・付加することなく、低コストで熱線遮蔽性を有する合わせガラスを実現することができる。

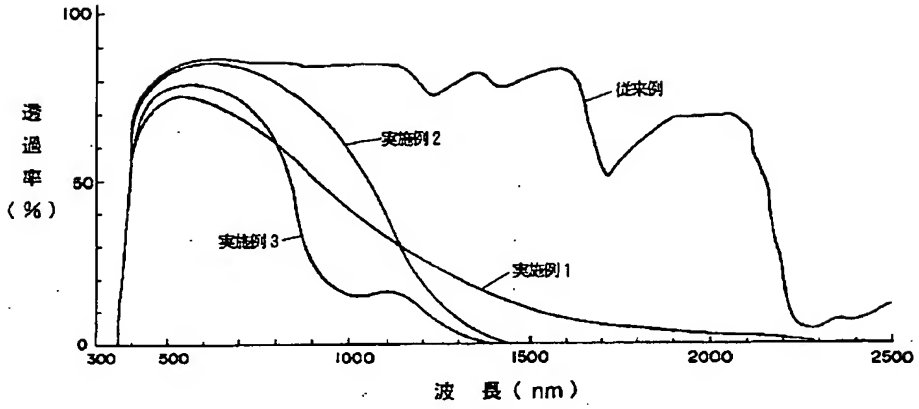
【００３４】そしてまた、請求項２記載の合わせガラスでは、前記熱線遮蔽性金属酸化物が粒径０．１μm以下の酸化スズまたは酸化インジウムのいずれかであるため、可視光の吸収が極力少なく、かつ熱線遮蔽性が良い合わせガラスを実現できる。

【００３５】そしてまた、請求項３記載の合わせガラスでは、前記熱線遮蔽性金属酸化物の配合比として０．４ｇ／m^2 以上となるように配合したため、可視光線透過率と日射透過率の差が５％以上にすることができる。

【図面の簡単な説明】

【図１】本発明の合わせガラスの光学特性を示すグラフである。

【図１】


100
50
0
透過率（%）
300
500
1000
1500
2000
2500
波長（nm）
従来例
実施例2
実施例3
実施例1